Investigation on Thermal Aging of Ester from Palm Oil and Kraft Paper Composite Insulation System for High Voltage Transformer

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Abstract: - Mineral oil is widely being used as insulation for high voltage equipments such as transformer due to its excellent dielectric properties. Recently, due to environment consideration and the long term availability, organic liquid insulations from plantations are being introduced. However, the performance of such insulations in composite with paper should be investigated in order to maintain the performances of high voltage equipments in the electric power system. This paper reports the experimental results on the effects of thermal aging on the properties of natural ester from palm oil and Kraft paper composite insulation.The samples used in these experiments were natural ester from palm oil and kraft paper transformer insulation. The ester samples of 800 ml and 6 gram of kraft insulation paper were put together in heat-resistant hermetic bottles to simulate actual condition of transformator.All samples were subjected to thermal aging condition for duration of 336 hours to 672 hours at 120^{o}C and 150^{o}C in a controllable oven. The properties of new sample as well as thermally aged samples were investigated. Dielectric properties such as breakdown voltage, resistivity, water content were measured and dissolved gas analysis were conducted. The chemical element change was measured using JEOL 6510 EDS (energy dispersive X-ray spectroscopy) with accelerated voltage up to 30 kV. The experimental results showed that thermal aging process greatly affected the breakdown voltage and resistivity. At initial stage of thermal aging application the breakdown voltage increased significantly from 33.2 kV to 43.9 kV due to the reduction of water content. At the later time, however, the breakdown voltage reduced due to the drastic increase of oxidation by product which was indicated by the darker color. Resistivity reduced drastically from 8.5 TΩ.cm to 1.3 TΩ.cm after aging at 150^{o}C for duration of 672 hour. Dissolved gas analysis indicated that thermal aging in ester-paper composite insulation released, CH_4 (Methane), C_2H_6 (ethane), and CO (Carbon Monoxide). The EDS analysis showed that during aging the C element increased while O element decreased. Tensile strength of paper reduced significantly with thermal aging. EDS data of Kraft paper aged at 120^{o}C for a duration of 4 weeks (672 hours) showed that the aged paper consists of 3 elements, C (75.23 %) , O (21.69 %) and K (3.08%). C was observed at energy of 0.277 keV, O at energy of 0.525 keV while K was observed at energy of 3.312 keV. Experimental results revealed that C increased with aging while O decreased with aging. On the other hand oxygen reduced from 41.17 % to 21.69 % because the oxygen from the paper reacted with oil in an oxidation and release CO gas as confirmed by DGA (dissolved gas analysis). Similar behavior was observed for aging at 150^{o}C.

Key-Words: -Palm oil, ester, Kraft paper, dielectric properties, tensile strength, thermal aging, high voltage transformer, DGA, EDS

1 Introduction

Due to constantly increase of electric power demand; the high voltage transmission system is widely used in transferring electric power form generating stations to the consumers. In an electric power system, high voltage equipments play important role. The reliability of the system is greatly dependent on the performances of the key equipments. In the high voltage equipment, insulation is one of the most important parts to withstand a high electric field during the operation of the equipments. Petroleum-based mineral oils have been used as composite insulation with Kraft paper for power transformer since long time ago. However, due to the environmental aspects and the availability in the future, researchers are searching new types of insulating materials, which are friendly to environment and renewable for the future availability. Some of natural esters were already introduced [1-5]. Natural ester shows a high breakdown voltage and flash point. In order to obtain better performances, mixture with mineral oil was also reported. Taking into account the requirements from standard, the mixture of natural ester from palm oil and mineral oil with ester content of 50 % is considered as good enough to be used as a high voltage insulating liquid. [5].

Investigation to apply the natural ester for distribution as well as high voltage power transformers were reported [6-10]. Natural ester from palm oil composed of hydro carbon of methyl and fatty acid and C-O (single bonding) and C=O (double bonding) of ester.

Kraft paper insulation is composed of approximately 90% of cellulose, 6-7% hemi-cellulose and 3-4% of lignin. A dry wood Kraft paper contains 40 to 50% of cellulose, 10-30% hemi-cellulose and about 20-30% lignin.

The chemical formula of cellulose that composes a paper is $(C_6H_{10}O_5)_n$ [8] .Typically it has density of 1.5 g/cm³. The chemical structure is shown in figure 1.

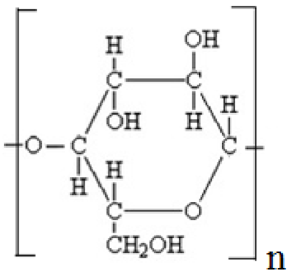


Figure 1. Chemical structure of cellulose

The number of n indicates the molecular size of the cellulose and is commonly called as the degree of polymerization (DP). DP strongly relates with the mechanical strength of the paper (tensile strength).

Thermal ageing can change the dielectric properties of insulation oil. Long term aging may finally causes the failure of the transformer leading to the interruption of power delivery.

In this paper, the experimental results on the thermal aging of Kraft paper-ester from palm oil composite insulation are presented. The thermal aging was conducted at 120 °C and 150 °C for duration 336 hours and 672 hours. The effects of the thermal aging to the dielectric properties and dissolved gas in ester are reported. The thermal aging effects on morphological as well as chemical change of Kraft paper also discussed.

The generated gases during the thermal aging are analysed using DGA (dissolved gas analysis) method [11-14]. The chemical element change was investigated using EDS (energy dispersive spectroscopy) [15-17].

2 Experimental setup

2.1 Sample

Samples used in this experiment were natural ester from palm oil and Kraft paper. The ester was made from palm oil trough an esterification using KOH as catalyst. The Kraft paper is widely used as high voltage transformer insulation. A set of sample was composed of 6 gram of dry paper insulation and 800 ml of ester. The oil and paper were put into heat-resistant and sealed glass bottle. A number of bottles with sample inside were placed in two ovens where their temperatures were kept constant at 120°C and 150°C to simulate an accelerated aging of the samples. 150°C temperature level selected by reference to research published in the IEEE by Mc Shane, et al [18], while the temperature of 120°C is a hot spot temperature according to the IEEE[19]. Example of the samples are shown in figure 1.

Paper insulation and oil samples were taken out at a regular interval of 336 hours to determine their dielectric properties and dissolved gasses in oil. Samples used with their aging treatments are shown in table 1.

(a) (b)

Fig.1 Sample (a) Ester and (b) paper

Table.1 Sample aging treatment

Sample	Condition
O	New (Initial State)
A1	Aging at 120°C for 336 hours
B1	Aging at 150°C for 336 hours
A2	Aging at 120°C for 672 hours
B2	Aging at 150°C for 672 hours

2.2 Measurement of Dielectric Properties

Dielectric properties such as breakdown voltage, resistivity and water content were measured before and after thermal aging. Breakdown voltage measurement was conducted in accordance with IEC 60156, while DC resistivity measurement was conducted using dielectric measurement system in accordance with IEC 60247 [20-21]. In this experiment the breakdown voltage was measured using bi-spherical standard cell with spacing of 2.5 mm in Liquid Dielectric Test Set LD60 from Phenix Technologies. AC voltage with frequency of 50 Hz was applied with increasing rate of 2 kV/s according to IEC 60156 until the breakdown of the liquid sample in the test cell. The breakdown voltage tester and the bi-spherical electrode are shown in figure 2.

(a) (b)

Fig. 2 The breakdown voltage tester (a) and the bi-spherical electrode (b)

Ester resistivity before and after aging were measured using high resistance meter. The measurements was conducted to oil sample with amount of 15 ml, and put in a standard liquid test cell from Tettex Instruments as shown in figure 3.

Figure 3. Liquid Test Cell and resistivity Meter

Water content measurement was done using Karl Fischer method according to ASTM D-1533 Standard [22]. This measurement was conducted using Karl Fischer KF875 as shown in figure 4.

Fig. 4 KF875 for determination of water content

2.3 Dissolved Gas Measurement

Due to thermal and or electrical stresses in the transformer, gasses may be generated in liquid insulation. In this experiment the generated gasses in ester from palm oil and paper composite due to thermal aging was measured using dissolved gases analysis system (DGA). Dissolved gases in the oil samples were measured using gas chromatograph HP 6890 integrated with Automatic Liquid Sampler HP 7649. The chromatograph is shown in figure 5. The concentration of combustible gasses i.e. H_2 (hydrogen), CH_4 (methane), C_2H_6 (ethane), C_2H_4 (ethylene) and C_2H_2 (acetylene) was determined using DGA(dissolved gas analysis). The procedure to extract the gas from the oil sample refers to the standard ANSI / IEEE C57.104 (1991) and ASTM D3612[23-24].

Oil samples were placed in a vial. Vials containing about 15 mlsample then placed in the sample container. An automated sample traction device takes the vial to be analysed. Vial is shaken by an automatic shaker to extract the gas trapped in the oil. Gases accumulated in the top of the vial, then collected automatically.

Fig. 5 Gas Chromatograph HP 6890 integrated with Sampler HP7649

The species of the dissolved gases and their concentration can be determined using the DGA.

2.4 Paper characterization

In order to understand the effects of thermal aging on the properties of Kraft paper in natural ester several measurements are conducted. They are visual observation, tensile strength measurement, scanning electron microscopy (SEM) and ED's measurement. The tensile strength of the paper was determined using tensile strength meter as shown in figure 6.

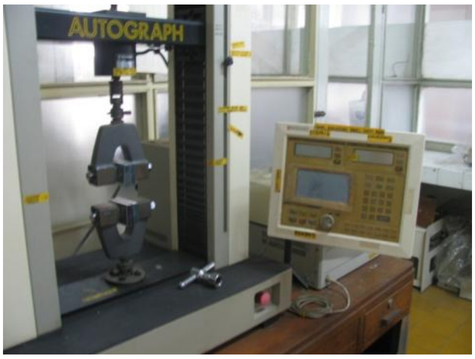


Fig. 6 Tensile strength measurement system

Morphology of paper was observed using SEM JEOL JSM 6610 series with accelerated voltage of 0.3-30 kV as shown in figure 7. The equipment has a built in EDS which enables to identify the chemical elements in the paper. The SEM and EDS measurement was conducted to the Kraft papers before and after thermal aging. This method is very useful to identify new chemical component responsible for corrosion in transformers [25].

Fig. 7 SEM JEOL JSM 6610

3 Experimental Results

3.1 Dielectric Properties of ester sample

Table 2 shows the visual appearance, breakdown voltage, resistivity and water content in oil before and after aging at 120^{o}C and 120^{o}C and aging time of 336 and 672 hours. The ester sample color became darker as aging became longer. Under thermal aging at 120^{o}C, the breakdown voltage increased from 33.2 kV to 43.9 kV at 336 hours and then decrease to 41.1 kV at 672 hours. The increase of breakdown voltage was due to the great reduction of water content from 1061.9 to 478.4 mg/kg. The breakdown voltage reduction at later period was due to contaminant from oxidation by products as indicated by very dark color of the sample. Later this will be confirmed from the dissolved gas analysis (DGA). Under thermal aging at 150^{o}C the similar behavior of breakdown voltage was observed.

Table 2
Dielectric properties of Ester sample before and after aging

Sample	**Visual appearance**	Breakdown voltage (kV)	Resistivity ($\times$ 10^{12}Ohm cm)	Water content (mg/kg)
New		33,2	8.5	1061,9
120^o 336h		43,9	7	478,4
120^o 672h		41,1	6	438,3
150^o 336h		39.75	1.5	931,86
150^o 672h		35.6	1.3	558,93

Fig. 8 shows the dependence of breakdown voltage of ester on ageing time at different aging temperature. The figure indicates that breakdown voltage of ester increased at 336 hours, then decreased after 672 hours. Under thermal ageing both of 120 ^{0}C and 150 ^{0}C, breakdown voltage in ester oil still much higher than standard IEC 60296 with limit of 30 kV. The increased of breakdown voltage at initial aging up to 336 hour was due to the significant reduction of water content from 1061,9 ppm to 478,4 ppm at aging temperature of 120^o C and to 438, 3 ppm at aging temperature of 150^o C as shown in table 2. However, at longer aging time the effect of water content reduction is much lower than the appearance of oxidation by product in the oil as indicated by the darker color of the sample at longer aging time. This leads to the decrease of the breakdown voltage.

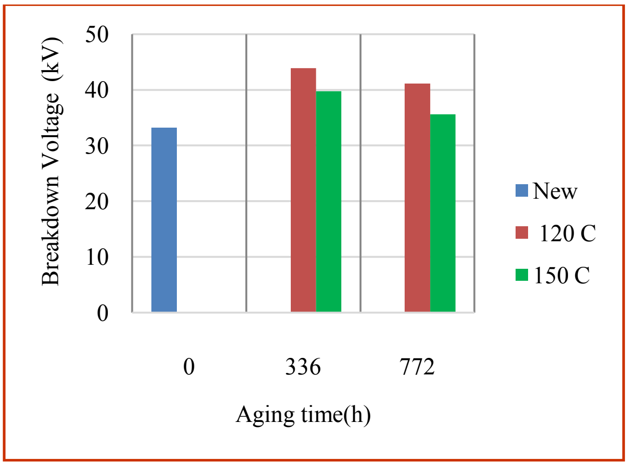


Fig.8 Dependence of breakdown voltage of ester on aging time at different aging temperature

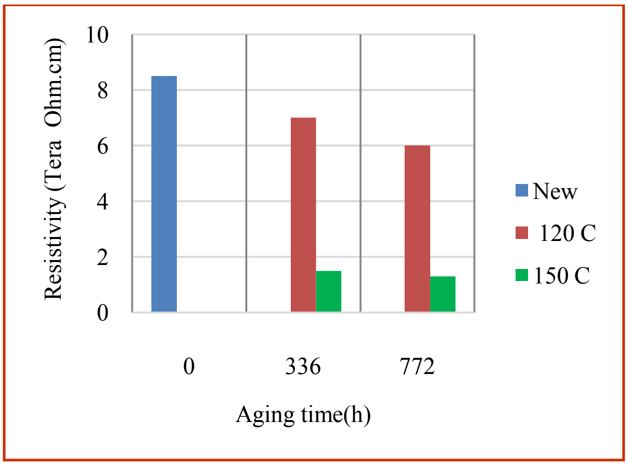


Fig.9 Dependence of resistivity of ester on aging time at different aging temperature

Figure 9 shown the dependence of resistivity on aging time at different aging temperature. The figure indicates that the resistivity of the ester from palm oil sample decreased with aging time and the aging temperature. Resistivity reduced drastically from 8.5 TΩ.cm to 1.3 TΩ.cm after aging at 150^{o}C for duration of 672 hour. The appearance of aging by product in the sample promotes the conduction of electron and thus increased the conductivity and reduced the resistivity.

3.2 Dissolved Gasses Analysis

Table 3 shows the concentration of combustible gases dissolved in the ester from palm samples aged with paper at different aging temperature and time. Detected combustible gases are H_2 (Hydrogen), CH_4 (Methane), C_2H_6 (ethane), and CO (Carbon Monoxide). CH_4 (Methane) was detected at the early aging while C_2H_6 (ethane) was detected at all aging temperature and time.

From the table 3 it is clear that CO appeared in all of the samples. At high-temperature cellulose molecules are decomposed and evolve carbon oxides (CO_2 and CO). High level of dissolved carbon oxides in oil indicates the thermal degradation of cellulose insulation in the system. Hence, it is possible to assess the level of solid insulation degradation using amount of CO2 and CO concentration in oil.

Table 3. DGA results on Ester from palm oil

Sample	Combustible Gas (ppm)					
	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO
0	0	0	0	0	0	0
A1	0	30,68	0	0	207,95	1674,58
B1	0	55,06	0	0	309,44	2264,89
A2	0	0	0	0	52,32	1135,28
B2	0	0	0	0	115,17	2159,24

The reason why C_2H_6 gas were generated in this experimental results considered to be the molecular bonding structures of natural ester fluid. Natural ester fluid is composed of unsaturated fatty acid structures which are compositely composed of double bonds and single bonds of carbon. When these structures are heated, the single bonds (C-C) that have a weaker binding force are degraded first to generate hydrocarbon. The hydrocarbon and water weaken the double bonds (C=C), so that the double bonds are decomposed to single bonds.

3.3 Effects of thermal aging on paper properties

3.3.1 Visual and tensile strength

Table 4 shows visual appearance and the % tensile strength as compared with the initial one.

Table 4 Visual appearance and tensile strength of paper in ester from palm oil

Sample number	Aging Condition	Visual appearance	*Tensile strength (%)*
0	New		100
A1	120^o 336h		88
A2	120^o 672h		60
B1	150^o 336h		80
B2	150^o 672h		50

From the table it is seen that the tensile strength reduced from 100 % at the beginning to 88 % after aging at 120^oC for 336 hours and then reduced to 60 % after aging period of 672 hours. At the aging tenperature of 150^oC , the tensile strength reduced to 80% after 336 hour aging and then dropped to 50 % after 672 hours. These facts clearly indicated that tensile strength of paper in ester from palm oil decrease with temperature and aging time. The decrease of tensile strenth due to thermal aging is clearly shown in figure 10.

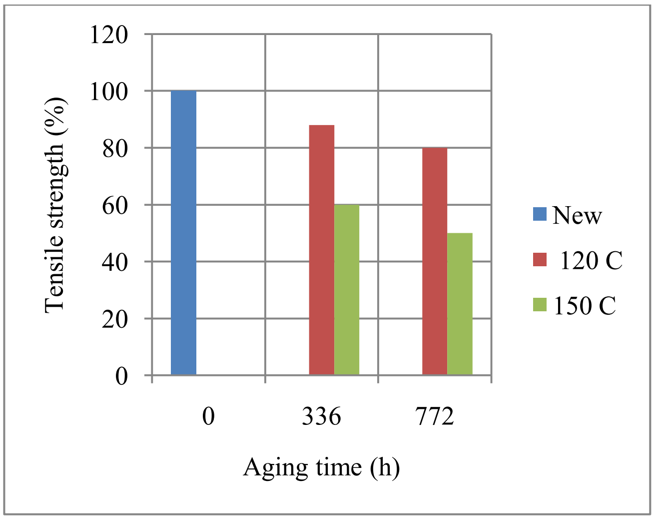
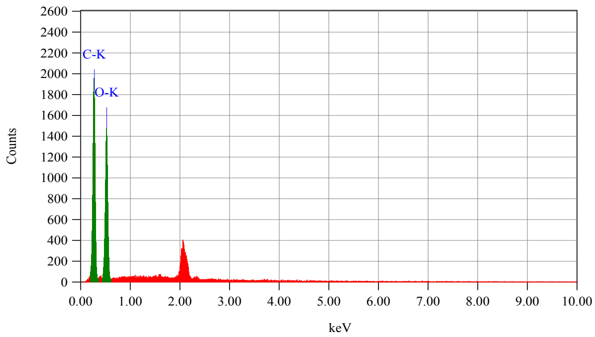


Fig. 10 Dependence of tensile strength of paper in Ester from palm oil on aging time and temperature

3.3.2 EDS result for new paper

Energy dispersive X-ray spectroscopy (EDS) measurement was conducted to identify the chemical element changes in the sample due to the thermal aging. The EDS uses X ray exposure with different level of energy (frequency) to identify the chemical elements in the sample. Each chemical element will show different response to the X ray exposure. Figure 11 shows the EDS spectrum taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 keV. From the spectrum it is seen that the new paper is mainly consists of C and O elements with mass percentage of 59.83 % of C at energy of 0.277 keV and O of 41.17 % observed at energy of 0.525 keV. The results are consistent with the facts that Paper is composed of Cellulose which is an organic compound with the formula n, a polysaccharide consisting of a linear chain of several hundred to many thousands of β linked D-glucose units. The chemical formula of cellulose is $(C_6H_{10}O_5)$



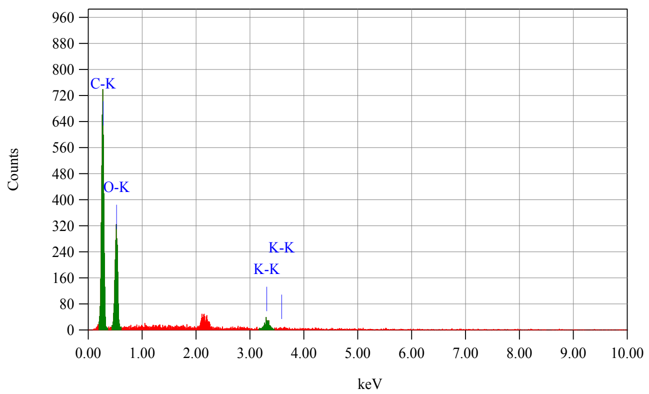
Fitting Coefficient : 0.6497

Element	(keV)	Mass%	Error%	Atom%
C K	0.277	58.83	0.23	65.55
O K	0.525	41.17	0.51	34.45
Total		100.00		100.00

Fig. 11 EDS spectra and chemical element for new paper

3.3.3 EDS result for paper aged at 120 deg for 336 hours in ester

Figure 12 shows the EDS spectrum and chemical elements for sample aged at 120°C for 336 hours. From the spectrum it is seen that the paper aged at 120°C for 336 hours in ester from palm oil mainly consists of C and O elements with mass percentage of 64.88 % of C at energy of 0.277 keV and O of 32.23 % observed at energy of 0.525 keV.



Fitting Coefficient : 0.7166

Element	(keV)	Mass%	Error%	Atom%
C K	0.277	64.88	0.26	72.11
O K	0.525	32.23	0.68	26.90
K K	3.312	2.89	1.18	0.99
Total		100.00		100.00

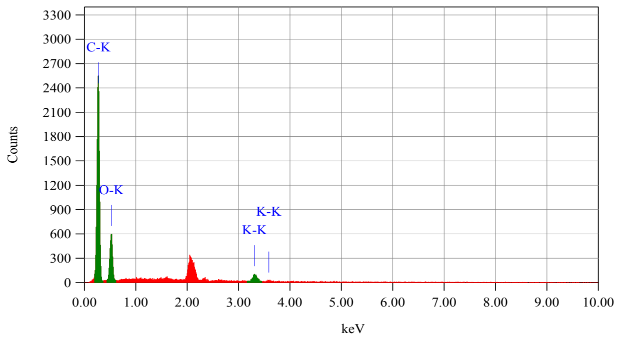
Fig. 12 EDS spectra and chemical element for paper sample aged at 120°C for 336 hours

The results indicates that the percentage of C increases while the percentage of O decreases. It is suggested that some of O from the paper structure was consumed and therefore the total amount reduced which implied to the increase of C percentage.

The experimental result also clearly indicated that new chemical element was found. It was K that observed at energy level of 3.312 keV. From the sample history it was found that the methyl ester from palm oil was made from palm oil trough an esterification process using KOH as catalyst and thus the K may come from the catalyst that reside in the sample.

3.3.4 EDS result for paper aged at 120 deg for 672 hours in ester

Figure 13 shows the EDS spectrum taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 keV.



Fitting Coefficient : 0.7659

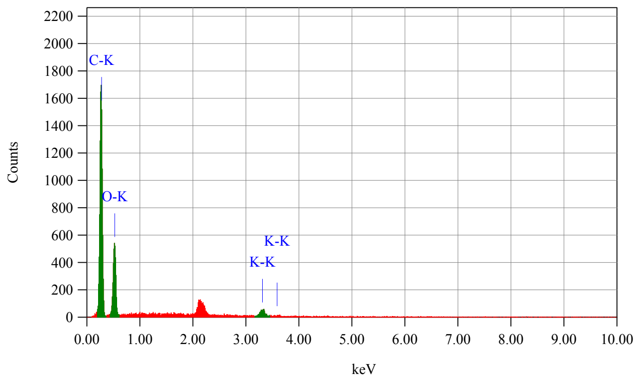
Element	(keV)	Mass%	Error%	Atom%
C K	0.277	75.23	0.29	81.37
O K	0.525	21.69	0.88	17.61
K K	3.312	3.08	1.35	1.02
Total		100.00		100.00

Fig.13 EDS spectra for paper in ester from palm oil aged at 120°C for 672 hours

From the spectrum it is seen that the aged paper consists of 3 elements, C , O and K elements with mass percentage of 75.23 % of C at energy of 0.277 keV , O of 21.69 % observed at energy of 0.525 keV and new element of K with mass percentage of 3.08 observed at energy of 3.312 keV. It is clearly observed that C increased from 58.83 % to 75.23 %. This is due to the migration from the ester into the paper on the other hand oxygen reduced from 41.17 % to 21.69 % because the oxygen from the paper reacted with oil in an oxidation and release CO gas as confirmed by DGA (dissolved gas analysis) which will be discussed later. From the EDS spectrum it is also seen that in the aged paper K element of 1.83 % mass is observed at energy of 3.312 keV. The sample used is a natural ester derived from palm oil. The main process is the transesterification reaction to get the targeted compound in the biodiesel product, the methyl ester. The Essential elements Reactants in this process are Methanol, Crude Palm Oil (CPO) and Alkali Catalyst KOH. The appearance of element K in the aged paper was due to the migration of K in the ester into the paper.

3.3.5 EDS result for paper aged at 150 deg for 336 hours in ester

Figure 14 shows the EDS spectrum and chemical elements for sample aged at 150^{o}C for 336 hours.



Fitting Coefficient : 0.7532

Element	(keV)	Mass%	Error%	Atom%
C K	0.277	70.73	0.28	77.20
O K	0.525	26.81	0.79	21.97
K K	3.312	2.46	1.30	0.82
Total		100.00		100.00

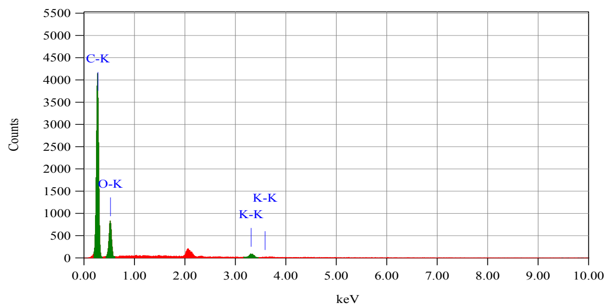
Fig.14 EDS spectra for paper in ester from palm oil aged at 150^{o}C for 336 hours

From the spectrum it is seen that the paper aged at 150^{o}C for 336 hours in ester from palm oil mainly consists of C and O elements with mass percentage of 70.73 % of C at energy of 0.277 keV and O of 26.81 % observed at energy of 0.525 keV. The results indicates that the percentage of C increases while the percentage of O decreases. The increase of C and the decrease of O were higher than that observed for the sample aged at 120^{o}C. The result suggested that the level of thermal aging plays important role in the percentage changes of C and O.

Similar with aging at 120oC, in this sample nwe chemical element was found. It was K that observed at energy level of 3.312 keV at the percentage of 2.46.

3.3.6 EDS result for paper aged at 150 deg for 672 hours in ester

Figure 15 shows the EDS spectrum taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 keV. From the spectrum it is seen that the aged paper consists of 3 elements, C, O and K elements with mass percentage of 78.19 % of C at energy of 0.277 keV, O of 19.98 % observed at energy of 0.525 keV and K with mass percentage of 1.83 % observed at energy of 3.312 keV.



Fitting Coefficient : 0.7748

Element	(keV)	Mass%	Error%	Atom%
C K	0.277	78.19	0.30	83.40
O K	0.525	19.98	0.94	16.00
K K	3.312	1.83	1.43	0.60
Total		100.00		100.00

Fig.15 EDS spectra for paper in ester from palm oil aged at 150^{o}C for 672 hours

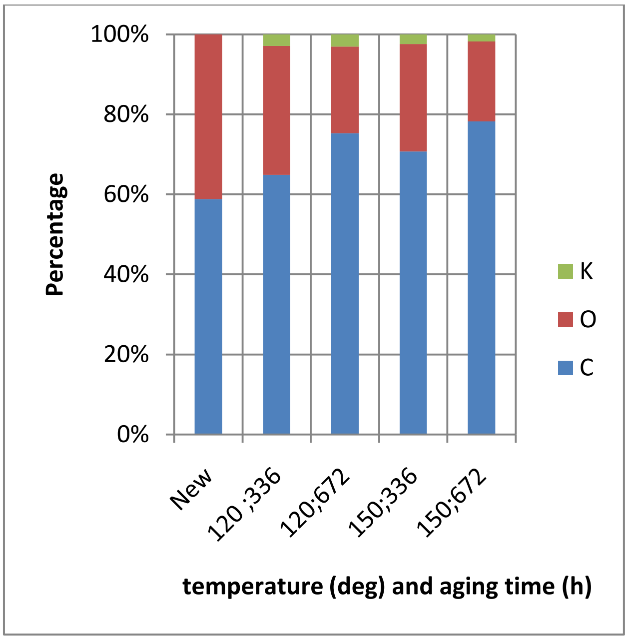
Thermal ageing of Kraft paper in ester oils yield mainly CO gas and its represent decomposition of cellulose. C_2H_6 gas generated from hydrocarbon bond that broke during ageing process, and small amount of H_2 and C_2H_2 due to different experimental ageing condition. Increasing temperature and the duration of the ageing proportional to total amount of dissolved gases. Gases that appear after ageing also affected breakdown voltage of ester oil, because gases had lower isolation capability than liquid, and hence, breakdown voltage of the ester oil decrease proportional to the ageing time.

The comparison of elements in new and aged Kraft paper samples is shown in table 5.

Table 5 comparison of elements in new and aged Kraft paper samples

Element	Energy (keV)	New (%)	Aged at 120°C for 336 h (%)	Aged at 120°C for 672 h (%)	Aged at 150°C for 336 h (%)	Aged at 150°C for 672 h (%)
C	0.277	58.83	64,88	75.23	70,73	78.19
O	0.525	41.17	32,23	21.69	26,81	19.98
K	3.312	0	2,89	3.08	2,46	1.83

From the table it is seen that the thermal aging in ester-Kraft paper composite insulation increased the C element due to ester permeation into the paper.



However, the thermal aging reduced the O element of paper because of the usage of oxygen element to oxidize the ester to release CO or CO2 which was confirmed by DGA. The small K element appeared after aging came from the catalyst during esterification

4 Conclusion

We have investigated the effects of thermal aging on natural ester from palm oil-Kraft paper composite system. The thermal aging was conducted at 120°C and 150°C for duration of 336hours and 672 hours respectively. The experimental results indicated that thermal aging process greatly affected the breakdown voltage and resistivity. Dissolved gas analysis indicated that thermal aging in ester-paper composite insulation released H_2 (Hydrogen), CH_4 (Methane), C_2H_2 (Acetylene), C_2H_4 (ethylene), C_2H_6 (ethane), and CO (Carbon Monoxide). The EDS analysis showed that during aging the C element increased while O element degreased. EDS data of Kraft paper aged at 120°C for a duration of 4 weeks (672 hours) taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 keV showed that the aged paper consists of 3 elements, C, O and K elements with mass percentage of 75.23 % of C at energy of 0.277 keV, O of 21.69 % observed at energy of 0.525 keV and new element of K with mass percentage of 3.08 observed at energy of 3.312 keV. It is clearly observed that C increased from 58.83 % to 75.23 %. This is due to the migration from the ester into the paper on the other hand oxygen reduced from 41.17 % to 21.69 % because the oxygen from the paper reacted with oil in an oxidation and release CO gas as confirmed by DGA (dissolved gas analysis). Similar behavior was observed for aging at 150°C. C element composition increased to 78.19 % of C at energy of 0.277 keV, while O element reduced to 19.98 % observed at energy of 0.525 keV and K with mass remain low at percentage of 1.83 % observed at energy of 3.312 keV. Thermal ageing of Kraft paper in ester oils yield mainly CO gas and this gas represents the decomposition of cellulose into the ester sample.

Acknowledgements

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